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## COSMETIC

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AbstractObjective

To provide cosmetics with excellent coverage of wrinkles, improved feel, natural finish, and sebum-adsorbing effect.

## Constitution

The cosmetics contain a modified silicone elastomer obtained from pulverization by continuous extrusion and kneading of the silicone elastomer using a continuous extrusion kneader.

## Claim

Cosmetics containing a modified silicone elastomer obtained from pulverization by continuous extrusion and kneading of the silicone elastomer, using a continuous extrusion kneader.

[0001]

### Technological field of the invention

The present invention pertains to cosmetics with excellent coverage of wrinkles, improved feel, natural finish, and sebum-adsorbing effect. More precisely, it pertains to cosmetics with excellent coverage of wrinkles, improved feel, natural finish, and sebum-adsorbing effect which can be obtained by compounding with modified silicone elastomer obtained from pulverization by continuous extrusion and kneading of the silicone elastomer, using a continuous extrusion kneader.

[0002]

### Prior art

As described in Japanese Patent Application No. Hei 8[1996]-52508, the present inventors found that solutions obtained by pulverizing silicon-containing resin powders with silicone oil had excellent coverage of wrinkles.

[0003]

### Problems to be solved by the invention

However, when solvents or oils are used, regardless of their volatility or nonvolatility, the effect of the solvents or oils on the cosmetic products is very large, especially in cosmetic powders such as foundations. For example, there were problems, such as the amount of silicone elastomers to be compounded having to be limited because of the amount of solvents or oils.

[0004]

On the other hand, when a silicone elastomer is directly compounded in cosmetic powders, etc., because the particles of the cosmetic powders form a hard mass, the surface of

molded cosmetics therefore becomes nonuniform; also, when liquid cosmetics containing silicone elastomer are filled in pump containers or aerosol containers and then they are discharged from the containers, there was the problem of the discharging tubes becoming clogged with particles of the cosmetics.

[0005]

#### Means to solve the problems

Therefore, the present inventors used various pulverization apparatuses to investigate direct compounding of silicone elastomers in cosmetics. For example, when an impact-fracturing type pulverizer such as an attriter, etc., was used, powders with a rubber-like elasticity, such as silicone elastomers, absorbed the impact force so that they could not be pulverized smoothly, and when a roller mill was used for kneading, the silicone elastomer could not be fed smoothly to the roller, therefore production was difficult.

[0006]

On the other hand, continuous extrusion kneaders, often used in food production, resin processing, etc., can pulverize powders while the powders are being fixed in a narrow space, therefore the present inventors thought that if a continuous extrusion kneader was used, it would be possible to pulverize silicone elastomers, which heretofore has been impossible. Therefore, a silicone elastomer was pulverized using a continuous extrusion kneader, such as a biaxial continuous extrusion kneader with removable block pattern for pulverizing, at 50°C, and as a result, it was found that aggregation of the powders was eliminated while the rubber-like elasticity of the silicone elastomer was maintained, and a modified silicone elastomer was obtained which had an excellent feel. And when it was compounded with cosmetics, it was found that cosmetics with excellent coverage of wrinkles, improved feel, natural finish, and sebum-adsorbing effect were obtained. Namely, the present invention is cosmetics obtained by compounding a modified silicone elastomer which was prepared from pulverization by continuous extrusion and kneading of the silicone elastomer using a continuous extrusion kneader.

[0007]

#### Embodiment of the invention

The silicone elastomer used in the present invention is a resin powder that has an average primary particle size, observed under an electron microscope, of preferably 0.1-20 µm, has a 3-dimensional crosslinking silicone structure, and may be used as a powder, as long as it has elastomer properties. Examples of such silicone elastomers include the Torayfil [transliteration]

E series products (Dow Corning Toray Silicone Co., Ltd.). In the above-mentioned silicone elastomers, those with few particles having a particle size greater than 12 µm are preferable because, when they contact the skin, there is little feeling of slipperiness.

[0008]

For the continuous extrusion kneader used in the present invention, the use of a multiaxial (at least biaxial) type continuous extrusion kneader which can perform continuous feeding and discharging with excellent kneading performance is preferable. For such a kneader, it is preferable to use a continuous extrusion kneader with an integrated assembly of extruding screws and a number of pulverizing block patterns whose arrangement can be changed to obtain specific kneading effects, rather than the common continuous extrusion kneader composed only of screws. Examples of the continuous extrusion kneader equipped with the pulverizing block patterns include extruders such as the SUPERTEX 30 extruder (Nippon Steel Corporation), etc. There is no limitation on the temperature during kneading, however 40-80°C is preferable, which is easy to control in mass production. When the kneading temperature is room temperature, sometimes it is difficult to control the temperature when the extrusion amount is increased.

[0009]

It is possible to install a mesh at the discharging outlet of the continuous extrusion kneader. However there have been cases in which the pulverized powders aggregated and granulated, so that the quality of the powders thus obtained were inferior, which is undesirable.

[0010]

Furthermore, the modified silicone elastomer obtained in the present invention may or may not undergo conventionally well-known surface treatments, for example, a fluorine compound treatment, such as perfluoroalkyl phosphate salt treatment, silicone treatment, silicone resin treatment, pendant treatment, silane coupling agent treatment, titanium coupling agent treatment, oil treatment, N-acylated lysine treatment, polyacrylic acid treatment, metallic soap treatment, amino acid treatment, inorganic compound treatment, plasma treatment, physicochemical treatment, etc. It is preferable to carry out such a surface treatment after the silicone elastomer has been modified. However, it is acceptable to carry out various surface treatments in advance.

[0011]

In the present invention, it is preferable to place the silicon elastomer alone in a continuous extrusion kneader. However it is also acceptable to mix the silicone elastomer with

fillers such as mica, sericite, talc, titanium oxide, zinc oxide, calcium carbonate, barium sulfate, polymethyl methacrylate, or hydroxyapatite, and then knead. In this case, like that mentioned above, the fillers may or may not undergo various surface treatments, however, from the viewpoint of compatibility with the silicone elastomer, undergoing a surface treatment with silicone, silicone resin, silica, and silane is preferred. In case it is to be mixed with a filler, the mixing ratio is such that the weight ratio of filler:silicone elastomer is from 0.1:99.9 to 90:10. If the weight ratio is in the above-mentioned range, an excellent feel of the silicone elastomer, as well as oil absorption, can be obtained remarkably. The preferable particle size of the above-mentioned fillers is such that the average primary particle size is 5 nm - 30  $\mu\text{m}$  and the particles are planar, spherical, needle-shaped, rod-shaped, spindle-shaped, amorphous, etc., thus, there is no limitation to the shape.

[0012]

The amount of modified silicone elastomer to be compounded in the cosmetics of the present invention is preferably 0.1-100 parts by weight, more preferably 1-60 parts by weight, based on 100 parts by weight of cosmetics. If in this range, excellent characteristics such as feel, oil absorption, etc., of the modified silicone elastomer can be obtained remarkably.

[0013]

In the cosmetics of the present invention, in addition to the above-mentioned ingredients, the following ingredients may be simultaneously compounded. Such additional ingredients which are usually used in regular cosmetics include oils, powders (pigment, coloring material, resin), fluorine compounds, resins, surfactants, tackifiers, preservatives, perfumes, ultraviolet ray absorbers (including organic or inorganic agents which absorb UV-A or UV-B), humectants, physiologically active ingredients, salts, solvents, antioxidants, chelating agents, neutralizing agents, pH-adjusting agents, etc.

[0014]

Examples of powders used in the present invention include coloring materials, such as Red No. 104, Red No. 201, Yellow No. 4, Blue No. 1, Black No. 401; color lake materials, such as Yellow No. 4 A1 Lake, Yellow No. 203 Barium Lake, etc.; polymers, such as nylon powder, silk powder, urethane powder, teflon powder, silicone powder, cellulose powder, etc.; color pigments, such as yellow iron oxide, red iron oxide, black iron oxide, chromium oxide, carbon black, ultramarine, prussian blue, etc.; white pigments, such as zinc oxide, titanium oxide, cerium oxide, etc.; extender pigments, such as talc, mica, sericite, kaolin, etc.; pearl pigments, such as mica titanium, etc.; metal salts, such as barium sulfate, calcium carbonate, magnesium

carbonate, aluminum silicate, magnesium silicate, etc.; inorganic powders, such as silica, alumina, etc.; titanium oxide fine particles, zinc oxide fine particles, iron oxide fine particles, alumina-treated titanium oxide fine particles, silica-treated titanium oxide fine particles, bentonite, smectite, etc. There is no limitation on the shapes of these powders.

[0015]

These powders may or may not undergo conventional surface treatment beforehand, such as a fluorine compound treatment, silicone treatment, silicone resin treatment, pendant treatment, silane coupling agent treatment, titanium coupling agent treatment, oil treatment, N-acylated lysine treatment, polyacrylic acid treatment, metallic soap treatment, amino acid treatment, inorganic compound treatment, plasma treatment, physicochemical treatment, etc. However, when carrying out a surface treatment, silicone treatment is most preferred.

[0016]

Examples of oils include higher alcohols, such as cetyl alcohol, isostearyl alcohol, lauryl alcohol, hexadecyl alcohol, octyldodecanol, etc.; fatty acids, such as isostearic acid, undecylenic acid, oleic acid, etc.; polyhydric alcohols, such as glycerin, sorbitol, ethylene glycol, propylene glycol, polyethylene glycol, etc.; esters, such as myristyl myristate, hexyl laurate, decyl oleate, isopropyl myristate, hexyldecyl dimethyloctanoate, monostearic acid glycerin, ethyl phthalate, monostearic acid ethylene glycol, octyl oxystearate, etc.; hydrocarbons, such as liquid paraffin, vaseline, squalane, etc.; waxes, such as hydrous lanolin, reduced hydrous lanolin, carnauba wax, etc.; fats and oils, such as mink oil, cocoa butter, coconut oil, palm kernel oil, camellia oil, sesame oil, castor oil, olive oil, etc.; ethylene- $\alpha$ -olefin cooligomer, etc.

[0017]

Examples of other forms of oil include silicone compounds, such as dimethylpolysiloxane, methylhydrogenpolysiloxane, methylphenylpolysiloxane, polyether-modified organopolysiloxanes, fluoroalkyl-polyoxyalkylene-comodified organopolysiloxanes, alkyl-modified organosiloxanes, terminal-modified organopolysiloxanes, fluorine-modified organopolysiloxanes, amodimethicone, amino-modified organopolysiloxanes, silicone gels, trimethylsiloxysilicic acid, silicone RTV rubbers, etc., and fluorine compounds, such as perfluoropolyether, fluorinated pitch, fluorocarbons, fluoroalcohols, etc.

[0018]

As surfactants, for example, anionic surfactants, cationic surfactants, nonionic surfactants, and amphoteric surfactants may be used.

[0019]

Examples of organic ultraviolet ray absorbers that can be used include 2-ethylhexyl p-methoxycinnamate, 2-hydroxy-4-methoxybenzophenone, 2-hydroxy-4-methoxybenzophenone-5-sulfuric acid, 2,2'-dihydroxy-4-methoxybenzophenone, p-methoxyhydrocinnamic acid, diethanolamine salt, p-aminobenzoic acid (hereinafter abbreviated to PABA), ethyldihydroxypropyl PABA, glyceryl PABA, homomenthyl salicylate, methyl-O-aminobenzoate, 2-ethylhexyl-2-cyano-3,3'-diphenyl acrylate, octyldimethyl PABA, octyl methoxycinnamate, octyl salicylate, 2-phenylbenzimidazole-5-sulfuric acid, salicylic acid triethanolamine, 3-(4-methylbenzilidene) camphor, 2,4-dihydroxybenzophenone, 2,2',4,4'-tetrahydroxybenzophenone, 2,2'-dihydroxy-4,4'-dimethoxybenzophenone, 2-hydroxy-4-N-octoxybenzophenone, 4-isopropyldibenzoylmethane, butylmethoxydibenzoylmethane, 4-ethylhexyl-4-(3,4-dimethoxyphenylmethylene)-2,5-dioxo-1-imidazolidine propionate.

[0020]

Examples of solvents that can be used in the present invention include purified water, cyclo silicone, ethanol, light liquid paraffin, lower alcohol, ethers, LPG, fluorocarbons, N-methylpyrrolidone, fluoroalcohols, volatile straight-chain silicones, etc.

[0021]

Examples of cosmetics of the present invention include makeup cosmetics such as concealer, foundation, white powder, rouge, lipstick, eye shadow, blush, nail color, filter color, etc.; hair cosmetics such as rinse, conditioner, hair treatment, etc.; basic cosmetics such as emulsion, lotion, cream, cleanser, pack, sunscreen agent, skin pore cover, aftershave lotion, preshave lotion, etc.; perfume, soap, deodorant, body powder, etc.

[0022]

#### Application examples

In the following, application examples and comparative examples will be used to explain the present invention in more detail. Methods used to evaluate properties of the cosmetics used in the application examples and comparative examples are shown below.

[0023]

(1) Effect of covering wrinkles

Cosmetics were applied to finely wrinkled skin around the eyes of middle-aged female panelists, then according to the standards of Table I, the wrinkle-covering effect was evaluated visually.

[0024]

Table I

| 基準                     | 判定 |
|------------------------|----|
| 小皺が目立つ ②               | ×  |
| 小皺がやや目立ちにくい ③          | △  |
| 小皺が目立たず、かつ見た目が不自然でない ④ | ○  |

Key:

- 1 Standard
- 2 Fine wrinkles are noticeable
- 3 Fine wrinkles are slightly noticeable
- 4 Fine wrinkles are not noticeable and the eyes are natural
- 5 Result of evaluation

[0025]

(2) Functional characteristic test

The feel when applying the cosmetics and the finish after applying the cosmetics were investigated by 10 specialists. According to the number of panelists who responded "excellent feel" and "natural finish," the cosmetics were evaluated according to the standards of Table II.

[0026]

Table II

| 基準(人数)<br>① | 判定<br>② |
|-------------|---------|
| 0~3名③       | ×       |
| 4~6名④       | △       |
| 7~10名⑤      | ○       |

Key:

- 1 Standard (number of panelist)
- 2 Result of evaluation
- 3 0-3 panelists
- 4 4-6 panelists
- 5 7-10 panelists

[0027]

Production Example 1 (modified silicone elastomer of the present invention)

As the silicone elastomer, "Torayfil E-701" (Dow Corning Toray Silicone Co., Ltd.) was used. As the continuous extrusion kneader, a SUPERTEX 30 (Nippon Steel Corporation) which jointly uses biaxial extrusion screws and block patterns for pulverization was used and the whole process was controlled at 50°C to carry out kneading and pulverization to give a modified silicone elastomer used in the present invention.

[0028]

Production Example 2 (modified silicone elastomer of the present invention)

As the silicone elastomer, "Torayfil-E 505C" (Dow Corning Toray Silicone Co., Ltd.) was used. As the continuous extrusion kneader, a SUPERTEX 30 extruder (Nippon Steel Corporation) which jointly uses biaxial extrusion screws and block patterns for pulverization was used to knead and pulverize a powder, prepared by mixing 70 parts by weight of silicone elastomer and 30 parts by weight of silicone-treated sericite using a Henschel mixer, and the entire process was controlled at 50°C to carry out kneading and pulverizing to give a modified silicone elastomer used in the present invention.

[0029]

Production Example 3 (modified silicone elastomer other than the present invention)

As the for silicone elastomer, "Torayfil E-701" (Dow Corning Toray Silicone Co., Ltd.) was used in a comparative example.

[0030]

Production Example 4 (modified silicone elastomer other than present invention)

As the silicone elastomer, "Torayfil E-701" (Dow Corning Toray Silicone Co., Ltd.) was pulverized using a high-speed flow-type mixing kneader (supermixer) which rotated mixing blades at high speed to carry out kneading and pulverization to give a silicone elastomer used in a comparative example other than the present invention.

[0031]

Production Example 5 (modified silicone elastomer other than the present invention)

As the silicone elastomer, "Trefil E 505C" (Dow Corning Toray Silicone Co., Ltd.) was used. 70 parts by weight of the silicone elastomer and 30 parts by weight of silicone-treated sericite were mixed, using a Henschel mixer, to give a silicone elastomer used as a comparative example other than the present invention.

[0032]

Application Example 1 (foundation of the present invention)

A foundation was prepared using the recipe shown in Table III. The unit of the compounding was "wt%." As the fluorine-treated pigment, 5 wt% a perfluoroalkyl phosphate ester salt-treated pigment was used.

[0033]

Table III

| 配合成分①                        | 配合量⑯ |
|------------------------------|------|
| 成分A ②                        |      |
| 改質シリコーンエラストマー (製造例1) ③       | 17   |
| フッ素処理セリサイト ④                 | 残量 ⑯ |
| フッ素処理タルク ⑤                   | 12   |
| フッ素処理酸化チタン ⑥                 | 10   |
| フッ素処理微粒子酸化チタン ⑦              | 5    |
| フッ素処理酸化鉄 ⑧                   | 4.5  |
| 成分B ⑨                        |      |
| スクワラン ⑩                      | 3    |
| ジメチルポリシロキサン ⑪                | 3    |
| フルオロアルキル・ポリオキシエチレン共変性シリコーン ⑫ | 3    |
| メチルフェニルポリシロキサン ⑬             | 1    |
| 防腐剤 ⑭                        | 0.2  |

Key:

- 1 Ingredient compounded
- 2 Ingredient A
- 3 Modified silicone elastomer (Production Example 1)
- 4 Fluorine-treated sericite
- 5 Fluorine-treated talc
- 6 Fluorine-treated titanium oxide
- 7 Fluorine-treated titanium oxide fine particles
- 8 Fluorine-treated iron oxide
- 9 Ingredient B
- 10 Squalane
- 11 Dimethylpolysiloxane
- 12 Fluoroalkyl-polyoxyethylene comodified silicone
- 13 Methylphenylpolysiloxane
- 14 Preservative
- 15 Amount compounded
- 16 Remainder

[0034]

Ingredients A were mixed in a mixer and, after adding the mixed solution of ingredients B, the resulting mixture was thoroughly mixed and the thus-obtained powder was molded to give a product.

[0035]

Application Example 2 (foundation of the present invention)

A foundation was prepared using recipe shown in Table IV. The unit was wt%. As the silicone-treated pigment, a 3 wt% of a methylhydrogenpolysiloxane-treated pigment was used, and as the fluorine-treated pigment, 5 wt% of perfluoroalkylphosphate ester salt-treated pigment was used.

[0036]

Table IV

| 配合成分 ①                     | 配合量 ⑯   |
|----------------------------|---------|
| 成分A ②                      |         |
| 改質シリコーンエラストマー (製造例2) ③     | 3 0     |
| シリコーン処理セリサイト ④             | 残量 ⑯    |
| シリコーン処理タルク ⑤               | 1 2     |
| シリコーン処理酸化チタン ⑥             | 1 0     |
| フッ素処理微粒子酸化チタン ⑦            | 5       |
| フッ素処理酸化鉄 ⑧                 | . 4 . 5 |
| 成分B ⑨                      |         |
| スクワラン ⑩                    | 2       |
| ジメチルポリシロキサン ⑪              | 1 . 4   |
| フルオロアルキル・ポリオキシエチレン共変性シリコーン | 1 0     |
| メチルフェニルポリシロキサン ⑬           | 1       |
| フッ素変性シリコーン樹脂 ⑭             | 0 . 6   |
| 防腐剤 ⑮                      | 0 . 2   |

|      |    |  |
|------|----|--|
| Key: | 1  | Ingredient compounded                              |
|      | 2  | Ingredient A                                       |
|      | 3  | Modified silicone elastomer (Production Example 2) |
|      | 4  | Silicone-treated sericite                          |
|      | 5  | Silicone-treated talc                              |
|      | 6  | Silicone-treated titanium oxide                    |
|      | 7  | Fluorine-treated titanium oxide fine particles     |
|      | 8  | Fluorine-treated iron oxide                        |
|      | 9  | Ingredient B                                       |
|      | 10 | Squalane   |
|      | 11 | Dimethylpolysiloxane                               |
|      | 12 | Fluoroalkyl-polyoxyethylene-comodified silicone    |
|      | 13 | Methylphenylpolysiloxane                           |
|      | 14 | Fluorine-modified silicone resin                   |
|      | 15 | Preservative                                       |
|      | 16 | Amount compounded                                  |
|      | 17 | Remainder  |

[0037]

Ingredients A were mixed in a mixer and, after adding the mixed solution of ingredients B, the resulting mixture was thoroughly mixed and the thus-obtained powder was molded to give a product.

[0038]

Comparative Example 1 (comparative example foundation)

The procedure of Application Example 1 was carried out, except that the silicone elastomer of Production Example 3 was used instead of the modified silicone elastomer of Application Example 1 to give a product.

[0039]

Comparative Example 2 comparative example foundation)

The procedure of Application Example 1 was carried out, except that the silicone elastomer of Example Production 4 was used instead of the modified silicone elastomer of Application Example 1 to give a product.

[0040]

Comparative Example 3 (comparative example foundation)

The procedure of Application Example 2 was carried out, except that the silicone elastomer of Manufacturing Example 5 was used instead of the modified silicone elastomer of Application Example 2 to give a product.

[0041]

Table V

|   | ① 錐の隠蔽効果 | 感触  | 仕上がり感 | 脂っぽさ |
|---|----------|--|-------|------|
|  実施例 1   | ○        | ○  | ○     | ○    |
|  実施例 2   | ○        | ○  | ○     | ○    |
|  比較例 1   | ×        | △  | ×     | △    |
|  比較例 2 | △        | △  | △     | △    |
|  比較例 3 | △        | △  | △     | △    |

Key:

- 1 Wrinkle-covering effect
- 2 Feeling
- 3 Finish
- 4 Oiliness
- 5 Application Example
- 6 Comparative Example

[0042]

From the results of Table V, we can see that the foundations of the application examples of the present invention received an excellent evaluation for all of the items tested, whereas there were problems in all of the items tested for the comparative example foundations. This is because the silicone elastomer in the comparative examples foundations was not loose in the products, so that it aggregated locally and became heterogeneous.

[0043]

#### Effect of the invention

From the above-mentioned results, it is clear that, by compounding a modified silicone elastomer obtained by kneading a silicone elastomer using a continuous extrusion kneader, cosmetics with excellent coverage of wrinkles, feel, natural finish, and sebum-adsorbing effect can be provided.